

METHOD STATEMENT

Determinand:

Metaldehyde

Matrix:

Sample Type: Treated, ground and raw water

Principle of Method:

The method is a large volume injection on-line solid phase extraction procedure. Samples are analysed by high performance liquid chromatography using a quadrupole time of flight mass spectrometer as a detector. The aqueous sample is injected by large volume injection (LVI) onto an enrichment column. The analyte is trapped on the enrichment column and then “back flushed” on to the analytical column. The analyte is identified and quantified by quadrupole time of flight mass spectrometric detection (MSD). Quantitation is based on an internal standardisation procedure.

Interferences:

HPLC-QTOF is an extremely selective technique and interferences should only be encountered very rarely. Any interfering compounds would have to display the identical QTOF transition at the same retention time; this is extremely unlikely in potable water samples. However, any compound, which passes through the extraction procedure, and has a similar liquid chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference. Samples containing high humic or fulvic loading have been demonstrated to not cause significant ion suppression for the compounds.

Performance of Method:

Range of Application:

LOD to 0.500 µg/l

Samples producing results above this range should be diluted and re-extracted

Limit of Detection:

0.005 µg/l

Recoveries of Compounds and Uncertainty of measurement:

Determinand	LOD ng/litre	Direct Standards				Elvington Treated Water (Hard Hardness)		
		Low Standard		High Standard		PCV Spike		
		% Recovery	RSD	% Recovery	RSD	% Recovery	RSD	Uncert
METALDEHYDE	3	107.3%	8.2%	98.8%	5.0%	99.4%	6.0%	± 12.5%

References:

Agilent 1200 Series, Reference Manuals.

Agilent 6400 QQQ LC/MS Techniques and Operation, Agilent Technologies Course Number R1893A, Student Manuals Volumes 1 and 2.

Time-of-Flight Mass Spectrometry, 5990-9207EN.

Agilent 6500 Series Q-TOF LC/MS system, Maintenance Guide, G2581-90065.

Agilent MassHunter Workstation Software Data Acquisition for 6200 Series TOF and 6500 Series Q-TOF, Familiarization Guide, G3335-90066

Agilent 6200 Series TOF and 6500 Series Q-TOF LC/MS System, Concept Guide, G3335-90142.

